

Soft X-ray Speckle and Dynamic Light Scattering Studies of Polystyrene Thin Films on BL12

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INTRODUCTION

A key goal of polymer physics is to forge a connection between microscopic fluctuations and macroscopic polymer properties. What microscopic forces conspire to determine that, at slow time scales, polymers are viscous, while at fast times scales they are elastic? Topological constraints - very likely entanglements of the polymer chains - play an important role in this problem. The important length scale is the so-called entanglement length, which measures the average separation between entanglements and is typically 4-8 nm. One way to study relatively slow fluctuations on this time scale is to apply the newly emerging techniques of dynamic x-ray or soft x-ray scattering. We have initiated a program in this area on BL12.0.1.2. Specifically, we wish to probe the thermally driven fluctuations at polymer surfaces at length scales as short as the entanglement length and to understand these in terms of other properties of the polymer.

APPARATUS

A schematic of the scattering apparatus is shown in Fig. 1. Light from the 8 cm period undulator passes through the BL12 varied line space monochromator, providing a beam with a resolving power of $E/\Delta E \sim 1000$ and a coherent fraction of a few per cent. Light exiting the monochromator is spatially filtered using a two-pinhole spatial filter that selects one transverse mode. At a wavelength of ~ 8 nm, we achieve a coherent flux as high as $2 \times 10^{10}/\text{sec}$. This light is then scattered off a sample and the scattered light is detected with a CCD camera.

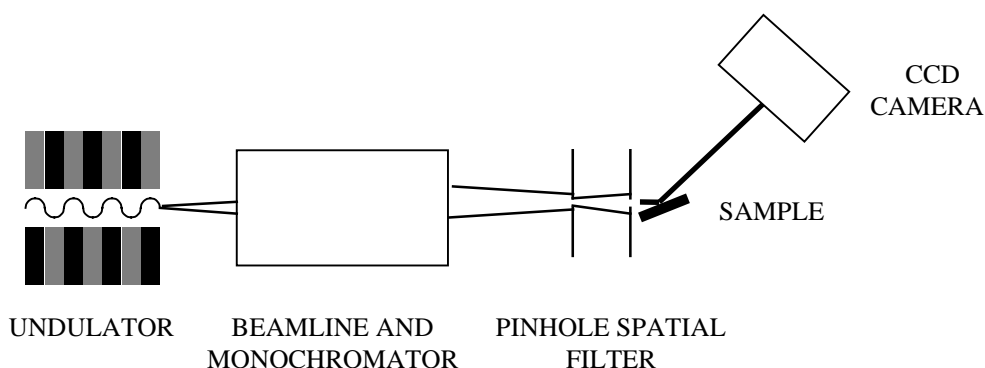


Figure 1: Schematic of the scattering apparatus, including the 8 cm undulator, the BL12 VLS monochromator, the spatial filter, the sample and CCD detector.

EXPERIMENTAL RESULTS

A precursor to the dynamic scattering experiment is to measure speckle-diffraction patterns from a static surface. An example is given in Fig. 2, which shows the distribution of light scattered in the near-specular direction from a high-quality polystyrene thin film surface. The left panel shows a gray-scale image, measured with the CCD camera, of transversely-coherent 75\AA light that has been scattered off a PS film at a grazing angle of 7.5° . The surface was held

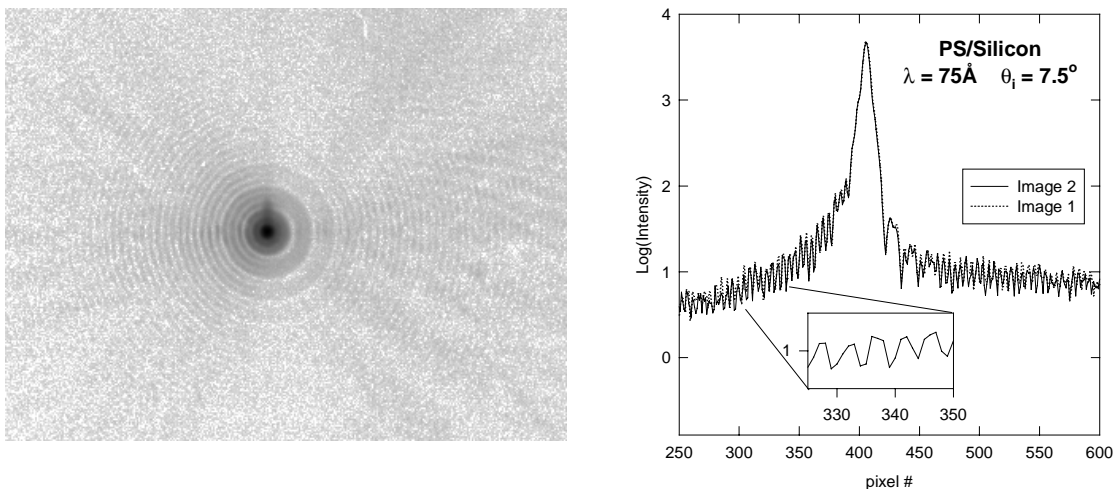


Fig. 2: Diffraction patterns observed upon scattering transversely-coherent 75\AA light off a high-quality polystyrene surface. left: gray-scale image; right: slice through the image showing the irregularity of the fringes caused by mixing of diffuse (speckled) light with the pinhole diffraction pattern.

at room temperature, which is well below the glass transition temperature, so that the surface was not fluctuating on the length scale probed by the experiment on the time scale required to take the image. The most obvious feature observed is the single-pinhole Airy function diffraction pattern associated with the pinhole spatial filter. The reason this appears is that the surface roughness is not sufficient to destroy the specular reflection, and the specular reflection in this case is this pinhole diffraction pattern. A closer look at this pattern indicates that the fringes are somewhat irregular. This is seen more clearly in the right panel of Fig. 2, which shows a single slice through the center of the image. The irregularity is seen to be reproducible from image to image, a notable characteristic of speckle. A slightly imperfect surface scatters most of the light specularly, with a small amount being diffusely scattered in the near-specular direction. If the incident light is transversely coherent, the diffuse light will be speckled, as approximately observed. Therefore, the image is actually a superposition of the pinhole diffraction pattern and the diffusely-scattered light - a weird sort of hologram. The former falls in intensity on a scale inversely related to the pinhole diameter ($10\text{ }\mu\text{m}$), while the latter decays in a fashion inversely related to the surface height - height correlation function (probably less than $1\text{ }\mu\text{m}$). We are currently working to understand these patterns in detail to determine whether they can be inverted directly.

More importantly, there is sufficient diffuse signal to show that dynamic scattering experiments will in principle be possible over a useful temporal dynamic range. This would entail measuring the intensity of the scattered light at a particular scattering angle and wave vector, and measuring the temporal autocorrelation function to deduce dynamical information of the corresponding length scale. We have initiated such studies and have found sample damage (both reversible and irreversible) to be a significant problem. Other scattering geometries and spatial filter configurations are being investigated to try to overcome these problems.

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